Effect of electrochemical treatments on the biodegradability of sanitary landfill leachates

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ABSTRACT

The application of combined electrocoagulation/anodic oxidation processes to improve the biodegradability of a leachate from an intermunicipal sanitary landfill was evaluated. Electrocoagulation (EC) experiments were performed with iron consumable electrodes, and the influence of the initial pH and of the electrocoagulation time were studied. In the anodic oxidation (AO) assays, a boron-doped diamond anode was used, and three applied current intensities were tested. The influence of the electrocoagulation pretreatment experimental conditions on the anodic oxidation performance was also evaluated. For the experimental conditions tested, there was an increase in the ratio between biochemical oxygen demand and chemical oxygen demand, BOD_5/COD, from 0.30 up to 0.88. The concentrations of iron, chromium and zinc were monitored during the combined treatment. During EC, chromium was almost completely removed, and zinc was partially removed; the remainder of the zinc was removed during AO. The concentration of iron increases during EC and decreases during AO; the removal of iron increases with the AO applied current density.

1. Introduction

Several types of pollutants can be found in the composition of sanitary landfill leachates, such as heavy metals, organic and inorganic compounds; some of these pollutants are refractory and toxic [1,2]. The composition and concentration of the pollutants are primarily influenced by the nature of the solid wastes deposited, the climatic conditions and the age of the sanitary landfill [3,4]. One of the major problems in sanitary landfill leachate treatment is the low biodegradability of the leachate, especially the mature leachates, because of the presence of recalcitrant and non-biodegradable organic substances [5,6]. Biological processes, used commonly to treat sanitary landfill leachates, have limited effectiveness when the ratio between the biological oxygen demand and the chemical oxygen demand, i.e., BOD_5/COD ratio, also known as the biodegradability index, is lower than 0.5 [3]. A possible solution for the treatment of sanitary landfill leachates with such properties is the use of an oxidation process as a pre-treatment to convert initially recalcitrant and non-biodegradable organic substances into more readily biodegradable intermediates [5,7,8].

Over the past 20 years, there have been several studies reporting the application of technologies based on oxidation processes to eliminate the color, reduce the organic load and, simultaneously, improve the biodegradability of the landfill leachates [4,5,9–19].

Yilmaz et al. studied the improvement in the BOD_5/COD ratio and the removal of COD and color from young municipal landfill leachate using the Fenton process [15]. The experimental results have shown a BOD_5/COD ratio increase from 0.58 to 0.64 and COD and color removals of 55.9% and 89.4%, respectively. An evaluation of the Fenton and the ozone-based advanced oxidation processes as a pre-treatment for mature landfill leachates showed that ozone in combination with hydrogen peroxide was the best oxidation approach tested, with COD removal reaching 72% and the BOD_5/COD ratio increasing from 0.01 to 0.24 [11]. A comparative study of the UV–Fenton, the UV–H_2O_2 and the Fenton reaction processes on landfill leachate was also reported, showing that the UV-Fenton process is the most effective approach to enhance the biodegradability index (from 0.17 to 0.60) and to eliminate the color of the leachate [16]. A solar photo-Fenton process combined with a biological nitrification and denitrification system was also proposed for the decontamination of a landfill leachate and was found to be efficient in the treatment of leachates, enhancing the biodegradability index and making possible a subsequent treatment by a biological oxidation process [17]. The application of a Fenton-like zero valent iron process on landfill leachates treatment was also tested, using iron shavings as the catalyst, which was shown to be a suitable and economical solution, leading to COD removals up to 60% [18].
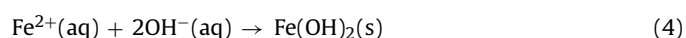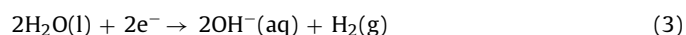
Another promising electrochemical method used in wastewater treatment is the anodic oxidation (AO) process [20–25]. Deng and Englehardt present an overview of the electrochemical oxidation processes used to treat landfill leachates [20]. Although different

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materials are being used as anodes in the oxidation of persistent pollutants, the best results are obtained with boron-doped diamond (BDD) anodes, due to their unique chemical, electrochemical and structural stabilities that allow their use at high potentials where most organic pollutants can be oxidized [26]. Zhao et al. proposed a synergistic combination of the biochemical treatment and the electrochemical oxidation of landfill leachate with sectional treatment on a BDD electrode that was highly efficient and energy saving, with biodegradability index improvements from 0.016 to 0.51, toxicity reductions from 82.5% to 30.2% and Total Organic Carbon (TOC) removals of 83.1% [27].

Another possible method of improving the biodegradability of sanitary landfill leachates is the removal of part of the recalcitrant and non-biodegradable organic substances. Although there are no reports in the literature that describe the use of electrocoagulation (EC) to improve the biodegradability of sanitary landfill leachates, several studies show that electrocoagulation is a promising method to remove heavy metals, arsenic, dyes and other persistent pollutants that were not sufficiently removed by conventional treatments [28]. In addition, EC techniques, using aluminium and iron electrodes, presented better performance than the chemical coagulation process in the treatment of landfill leachate, showing that it can be successfully used as part of a combined treatment [29].

In EC processes, the coagulating ions are produced 'in situ' and they are involved in three successive stages [30]: (i) formation of the coagulants by electrolytic oxidation of the 'sacrificial electrode', (ii) destabilization of the contaminants, particulate suspension and breaking of emulsions and (iii) aggregation of the destabilized phases to form flocs. The generation of metallic ions involved in the first stage of EC is dependent on the applied potential/current intensity and on the pH and the conductivity characteristics of the wastewater. The main processes that occur in the electrolytic system when iron anodes are used can be described by the reactions (1)–(4). At the anode, the Fe^{2+} is formed due to the oxidation of Fe, according to reaction (1). At the cathode, H_2 gas is formed from the reduction of the protons in the acidic medium (reaction (2)) or from water reduction in the alkaline medium (reaction (3)). In both cases, the pH increases during electrolysis, and the Fe^{2+} ions can react to form iron hydroxides (reaction (4)).

$$Fe(s) \rightarrow Fe^{2+}(aq) + 2e^- \qquad (1)$$

$$2H^+(aq) + 2e^- \rightarrow H_2(g) \qquad (2)$$

$$2H_2O(l) + 2e^- \rightarrow 2OH^-(aq) + H_2(g) \qquad (3)$$

$$Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s) \qquad (4)$$

In solution, iron ions hydrolyze and, depending on the medium pH, different polymeric hydroxides may be formed. The consumable iron anodes are used to continuously produce polymeric hydroxides close to the anode, which are excellent coagulation agents and have a strong affinity for colloids, dispersed particles and ionic species and cause flocculation. The formed flocs can be removed by sedimentation or flotation [28,31]. In the case of complex mixtures containing organic and inorganic matter, such as sanitary landfill leachates, simultaneously to the process described above, the cathodic reduction of the metal ions present in the suspension may also occur in the EC cell, with the consequent deposition of metals over the cathode.

The generally low biodegradability of the leachates from sanitary landfills shows that their biological treatment typically requires an extra aid, with chemical or physical pre-treatment or post-treatment procedures. The aim of this study was to evaluate the application of electrocoagulation and anodic oxidation in

a combined process for the improvement of the biodegradability of a leachate from an intermunicipal sanitary landfill. During EC treatment, large quantities of iron are introduced into the suspension; therefore, the iron concentration was also monitored during the combined treatment, as well as the concentrations of chromium and zinc, which were already present in the leachate samples used in this study.

2. Experimental

2.1. Analytical methods

Degradation tests were followed by chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), dissolved organic carbon (DOC), total nitrogen (TN), total Kjeldahl nitrogen (TKN) and ammonia nitrogen (AN) determinations, which were performed according to the standard methods [32]. COD determinations were made using the closed reflux titrimetric method. The BOD_5 was evaluated by determining the oxygen consumption after 5 days of incubation. The DOC and TN were measured in a Shimadzu TOC-V_{CPH} analyzer combined with a TNM-1 unit. Before DOC and TN determinations, samples were filtered through 1.2 μm glass microfiber filters. The TKN and AN were determined according to standard procedures using a Kjeldatherm block-digestion-system and a Vapodest 20 s distillation system, both from Gerhardt. The evolution of the concentration of iron, chromium and zinc during EC and AO was determined by flame atomic absorption spectrometry using a Perkin Elmer Apparatus. The sample preparation followed a standard procedure that includes an HCl–HNO_3 acid digestion [32].

2.2. Leachate characterization

The leachate samples used in this study were collected at a Portuguese intermunicipal sanitary landfill site. This site serves a population of over 220,000 and has an onsite facility capable of treating up to 50 m^3/day of leachate. The treatment applied at this landfill site comprises a biological step followed by an ultrafiltration operation. The raw leachate samples used in this study were collected in the equalization tank before the biological treatment, and their physicochemical characteristics are presented in Table 1.

2.3. Electrochemical experiments

The electrocoagulation experiments were conducted in batch mode, without stirring and using 500 mL of leachate. Iron consumable electrodes with an immersed area of 40 cm^2 were used as the anode and the cathode, with a gap between them of 1.0 cm. The experiments were conducted at an applied current intensity of 2.5 A, at room temperature (22–25 °C) and without addition of a background electrolyte. The two initial pH conditions studied were the natural pH (8.5) and a pH of 6. The influence of the EC assay

Table 1
Physicochemical characteristics of the raw leachate.

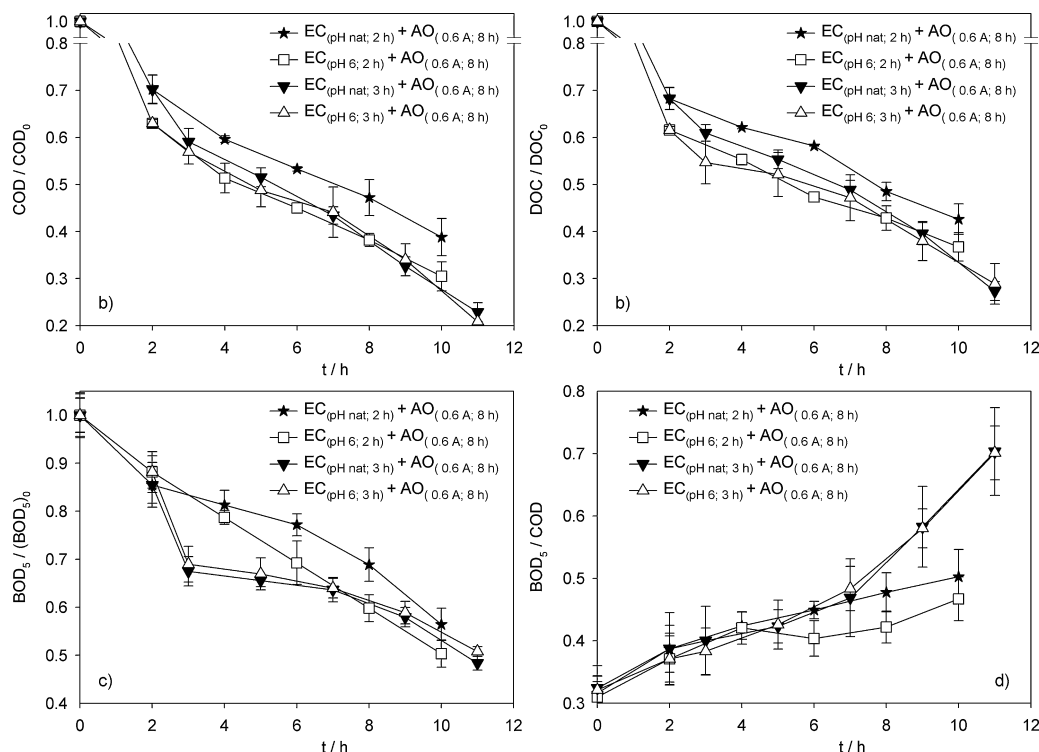| Property	Medium value (±SD[a])
COD/g L^{-1}	21.7 ± 0.5
BOD_5/g L^{-1}	6.5 ± 0.5
BOD_5/COD	0.30 ± 0.03
DOC/g L^{-1}	8.5 ± 0.2
TN/g L^{-1}	2.1 ± 0.1
TKN/g L^{-1}	2.0 ± 0.1
AN/g L^{-1}	1.2 ± 0.1
Chloride/g L^{-1}	4.4 ± 0.1
pH	8.5 ± 0.1
Conductivity/mS cm^{-1}	36.1 ± 0.4

[a] SD – standard deviation.

Fig. 1. Variation with time of the normalized (a) COD, (b) DOC and (c) BOD_5 and of the (d) BOD_5/COD ratio for the EC + AO experiments. EC conditions: initial pH of 6 or natural pH (8.5), 2 or 3 h duration, 2.5 A current intensity; AO conditions: 0.6 A current intensity, 8 h duration. Error bars refer to the standard deviation of the normalized mean values.

duration was also evaluated, using timed assays of 2 and 3 h. After the EC treatment, the samples were allowed to settle, and the supernatant liquid was submitted to the anodic oxidation treatment.

The anodic oxidation experiments were conducted in batch mode with recirculation, using 200 mL of the electrocoagulated effluent. A BDD anode and a stainless steel cathode, both with an area of 20 cm^2, were used. The current intensities evaluated were 0.6, 1 and 1.4 A. The electrochemical experiments employed a DC power supply GW, Lab DC, model GPS-3030D (0–30 V, 0–3 A). The recirculation of the sample was enabled by a centrifugal pump, Pan World Magnet, Model: NH-30PX, Pan World Co., Ltd. Tokyo, Japan, and the flow rate was kept constant at 75 L h^{-1}.

Adjustments to the pH were made by the addition of concentrated H_2SO_4 or NaOH solutions. The assays were performed in triplicate, and the values presented for the parameters used to follow the assays are the mean values.

3. Results and discussion

3.1. Influence of the EC pretreatment

Fig. 1 shows the results for the variation with time of the normalized COD, DOC and BOD_5 and of the BOD_5/COD ratio for the experiments performed with leachate under the following experimental conditions: EC at initial pH of 6 or the natural pH (8.5), duration of 2 or 3 h, at an applied current intensity of 2.5 A, followed by AO, for an 8 h duration at 0.6 A.

According to the literature, the pH of the solution plays an important role in the EC treatments [12]. An initial pH of 6 enhances the COD and DOC removal rates during the first 2 h of the electrocoagulation process. For the assays performed during 3 h of electrocoagulation, the results show a decrease in the COD and DOC removal rates during the third hour, indicating a loss of efficiency of the process during this extra hour of electrocoagulation. Despite the efficiency loss of COD and DOC removal, when electrocoagulation

is extended one more hour, the amount of organic load removed at the end of the combined process is higher, indicating that an extra hour of electrocoagulation benefits the combined process.

According to these results, and concerning COD and DOC removal, an initial pH of 6 is favorable for the combined process performance when 2 h of electrocoagulation are performed. However, for the experiments with 3 h of electrocoagulation, the effect of the pH is not significant, since the amount of COD that was removed after these 3 h remains essentially unchanged for both of the initial pH values studied.

Analysis of the BOD_5 results (Fig. 1c) shows that the third hour of electrocoagulation greatly increases the BOD_5 removal. As a consequence, the BOD_5 removal rate during the anodic oxidation process is much lower than the one presented by the assays performed with 2 h of electrocoagulation and at the end of the combined process, no significant differences can be found in the BOD_5 removals in the assays performed. An initial pH of 6 is favorable in the assays with 2 h of electrocoagulation, but no influence is found when 3 h of electrocoagulation are performed.

Regarding the BOD_5/COD ratio (Fig. 1d), even though there is no significant difference during the electrocoagulation process, at the end of the combined processes the assays performed with 3 h of electrocoagulation show higher BOD_5/COD ratios, indicating an important enhancement on the biodegradability index. For the assays performed with a 3 h EC duration, no significant influence of the initial pH is found. For the combined processes with a 2 h EC duration, adjusting the initial pH to 6 slightly reduces the final BOD_5/COD ratio. In spite of lower BOD_5 and COD removals in the combined process using the initial natural pH, the biodegradability index becomes favorable.

Fig. 2 shows the results obtained for the nitrogen removal. In the assays performed with 2 h of electrocoagulation, the initial pH of 6 does not promote enhancement of the removal of any nitrogen forms compared with assays using an initial natural pH. When the initial pH is adjusted to 6, ammonia nitrogen is formed during the
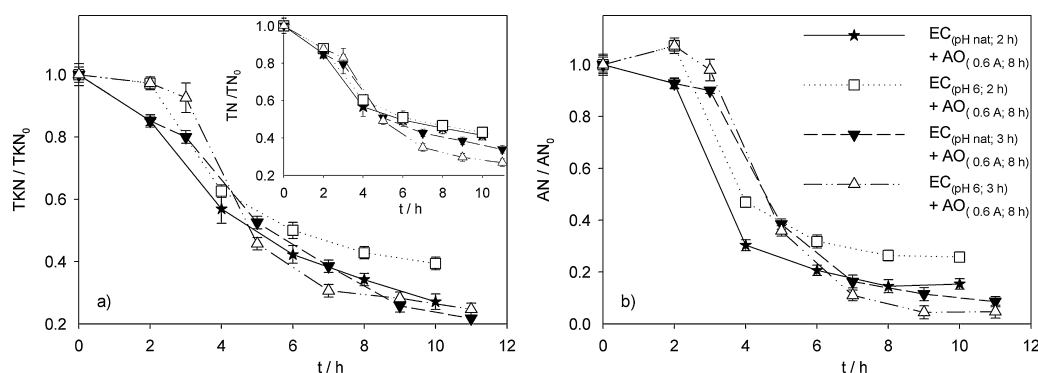
Fig. 2. Variation with time of the normalized (a) TKN, TN (inset) and (b) AN for the EC + AO experiments. EC conditions: initial pH of 6 or natural pH (8.5), 2 or 3 h duration, 2.5 A current intensity; AO conditions: 0.6 A current intensity, 8 h duration. Error bars refer to the standard deviation of the normalized mean values.

first 2 h of the electrocoagulation process, leading to an increase in AN levels and a low removal of TKN. Although electrocoagulation is here the primary mechanism, simultaneous anodic oxidation and cathodic reduction of the species present in the leachate cannot be excluded and, in fact, similar formation of ammonia nitrogen from organic nitrogen was already reported during anodic oxidation in previous works [33,34], being responsible for the low removal of TKN, since TKN comprises organic and ammonia nitrogen forms. Regarding the higher AN levels attained during EC performed at pH of 6 when compared to natural pH, it must be due to the higher NH_4^+/NH_3 ratio, observed in acidic conditions, that reduces the elimination of nitrogen as NH_3 by stripping.

During the EC process with an initial pH of 6, the level of TN decreases due to the elimination of the inorganic and organic forms of nitrogen. Extending the electrocoagulation for one more hour improves the nitrogen removals, especially that of ammonium nitrogen, at the end of the combined process. The improvement in nitrogen removal is related to the COD removal, which is also

higher in these assays. For the chloride concentration present in the leachate samples used in this study, the ammonium removal usually occurs at a slower rate than the COD removal. The use of BDD anodes promotes the generation of hydroxyl radicals, while the high content of chloride induces the simultaneous formation of free chlorine, which is responsible for the indirect oxidation of ammonium [24]. Chlorine evolution is enhanced at lower COD concentrations, thus causing indirect oxidation of ammonium.

3.2. Influence of the AO current intensity

The pretreatment, consisting of 3 h of electrocoagulation at the natural initial pH, was performed to obtain samples to study the effect of the AO applied current intensity on the combined treatment.

The three different current intensities evaluated were 0.6, 1.0 and 1.4 A. Fig. 3a and b shows the normalized COD and DOC variations with time and with applied charge for these assays.
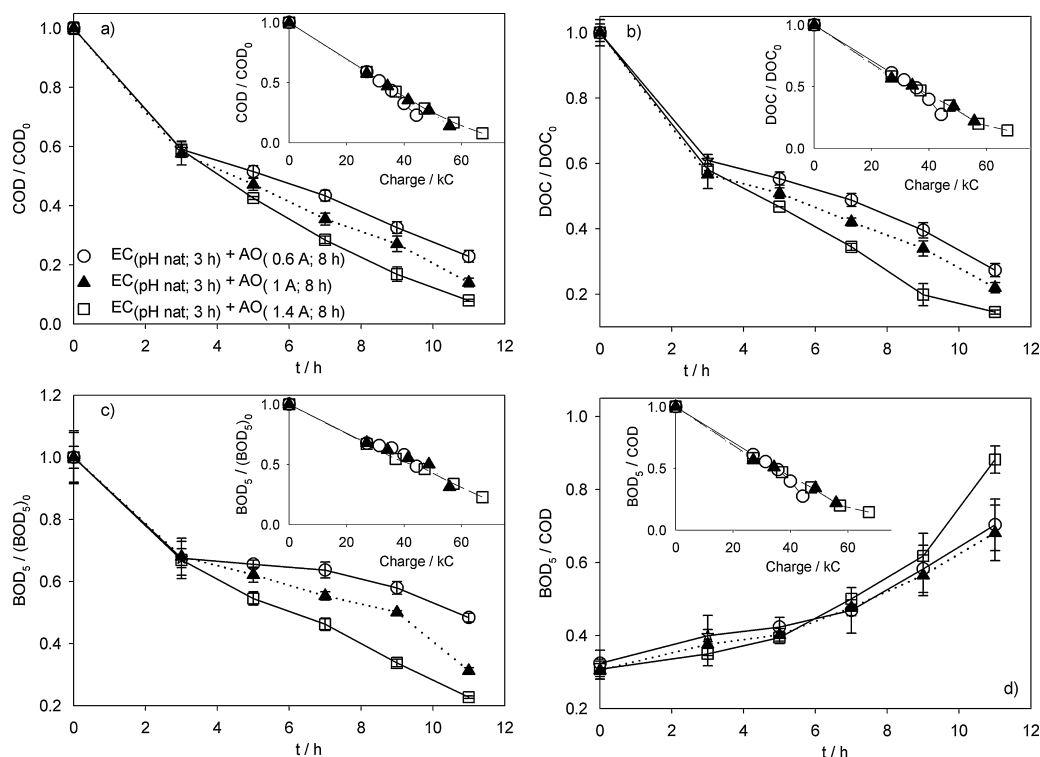

Fig. 3. Variation with time and with applied charge (insets) of the normalized (a) COD, (b) DOC and (c) BOD_5 and of the (d) BOD_5/COD ratio for the EC + AO experiments. EC conditions: natural initial pH, 3 h duration, 2.5 A current intensity; AO conditions: current intensities of 0.6 A, 1 A and 1.4 A, 8 h duration. Error bars refer to the standard deviation of the normalized mean values.
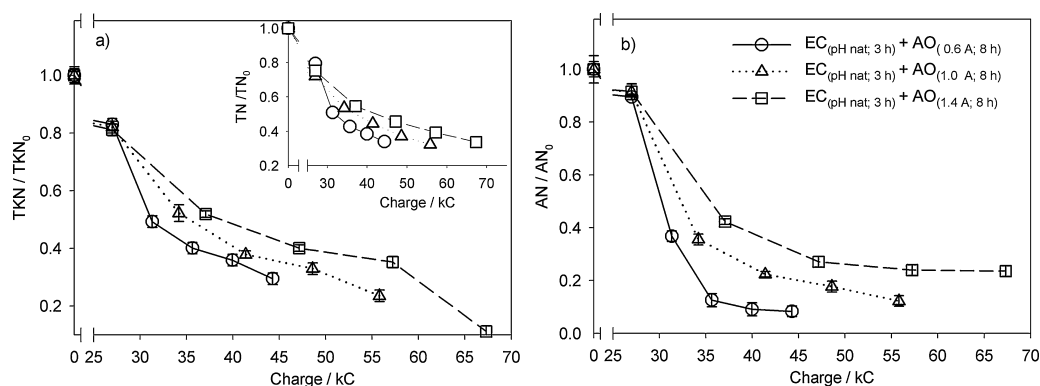
Fig. 4. Variation of the normalized (a) TKN, TN (inset) and (b) AN with the applied charge for the EC + AO experiments. EC conditions: natural initial pH, 3 h duration, 2.5 A current intensity; AO conditions: current intensities of 0.6 A, 1 A and 1.4 A, 8 h duration. Error bars refer to the standard deviation of the normalized mean values.

Both COD and DOC removal rates increase with applied current intensity during the anodic oxidation process, indicating that the process is operating under kinetic control, since COD values are higher than critical COD during most part of the assays duration, according to medium mass transfer coefficients presented in literature for leachate samples [23,25].

In terms of applied charge, for the applied current intensity of 0.6 A, the COD and DOC removal rates are higher, and the removal rates for the other two current intensities assayed were identical. This result is due to a higher current efficiency for the lowest current density. The BOD_5 and BOD_5/COD ratio behavior during the assays are also shown in Fig. 3. For the variations in time, it can be observed that the BOD_5 removal rate and the BOD_5/COD ratio are higher for the highest current intensity. However, if these parameters are plotted against the electric charge passed, then the observed differences are small, showing that the efficiency of the different assays is similar due to the high organic load of the samples under study. The best result for the BOD_5/COD ratio, 0.88, was achieved with an applied current intensity of 1.4 A after 8 h of anodic oxidation.

Fig. 4 shows the results obtained for nitrogen removal at the different intensities of the applied current. For all forms of the nitrogen measured, at the same applied charge, the higher applied current intensities are associated with lower nitrogen removal rates. These results are not in agreement with some reported in literature [21,23,24], but leachates used in this work possess much higher organic load content and were subjected to an electrocoagulation pretreatment, which may justify this different behavior.

3.3. Energy consumption

To analyze the effect of the different experimental conditions on the energy consumption, the instantaneous specific energy consumption, IEsp, in $Wh\,(gCOD)^{-1}$, was calculated using the following equation:

$$IEsp = \frac{1}{V_s}\frac{\partial E}{\partial(\Delta COD)} = \frac{1}{V_s}\frac{\partial E/\partial t}{\partial(\Delta COD)/\partial t} \tag{5}$$

where $E(=UIt)$ is the energy consumption in Wh, U is the cell voltage in V, resulting from the applied current intensity I in A, t is the duration of the electrolysis in h, ΔCOD is the COD removed in $g\,m^{-3}$ for the elapsed time and V_s is the volume of the solution in m^3. Determination of $\partial E/\partial t$ and $\partial(\Delta COD)/\partial t$ was performed by adjusting the polynomial equations to the variations of E and ΔCOD with time, and calculating the corresponding derivatives. Fig. 5(a) reports the instantaneous specific energy consumption as a function of time for the assays performed with different initial EC experimental conditions. Although the energy consumption during the electrocoagulation process at the natural pH is higher for the assays with a 3 h duration, it leads to an extra reduction in the specific energy consumption during the anodic oxidation process, indicating that the extra hour of EC can be beneficial for the entire combined process. However, for these two assays the medium specific energy consumption, Esp, determined as the ratio between the total energy consumption and the total COD removed, is almost equal: 15.1 and 15.4 $Wh\,(g\,COD)^{-1}$, for a 2 and 3 h EC pretreatment, respectively. For the assays performed with 2 h of electrocoagulation, the adjustment of the initial pH to 6 promoted a higher organic
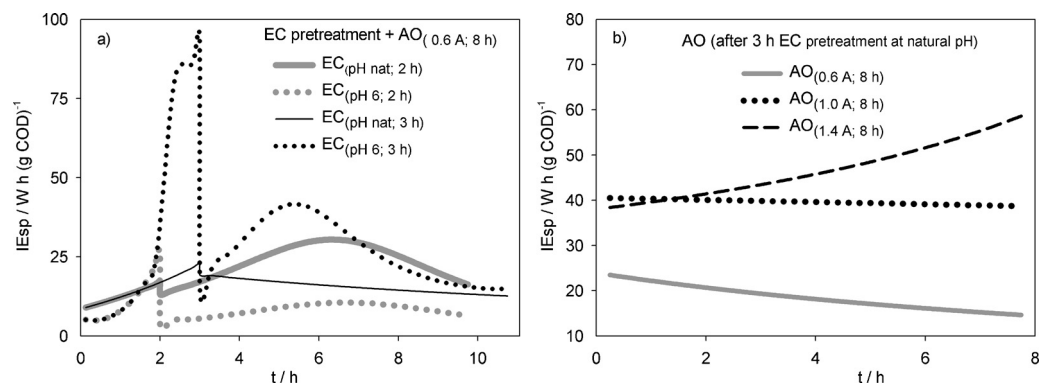

Fig. 5. Instantaneous specific energy consumption, IEsp, for the assays performed under the following experimental conditions: (a) EC at an initial pH of 6 or the natural pH (8.5), 2 or 3 h duration, at an applied current intensity of 2.5 A; AO at a current intensity of 0.6 A, 8 h duration; and (b) AO at a current intensity of 0.6 A, 1 A or 1.4 A, 8 h duration, after 3 h of EC pretreatment, at the natural pH, at an applied current intensity of 2.5 A.
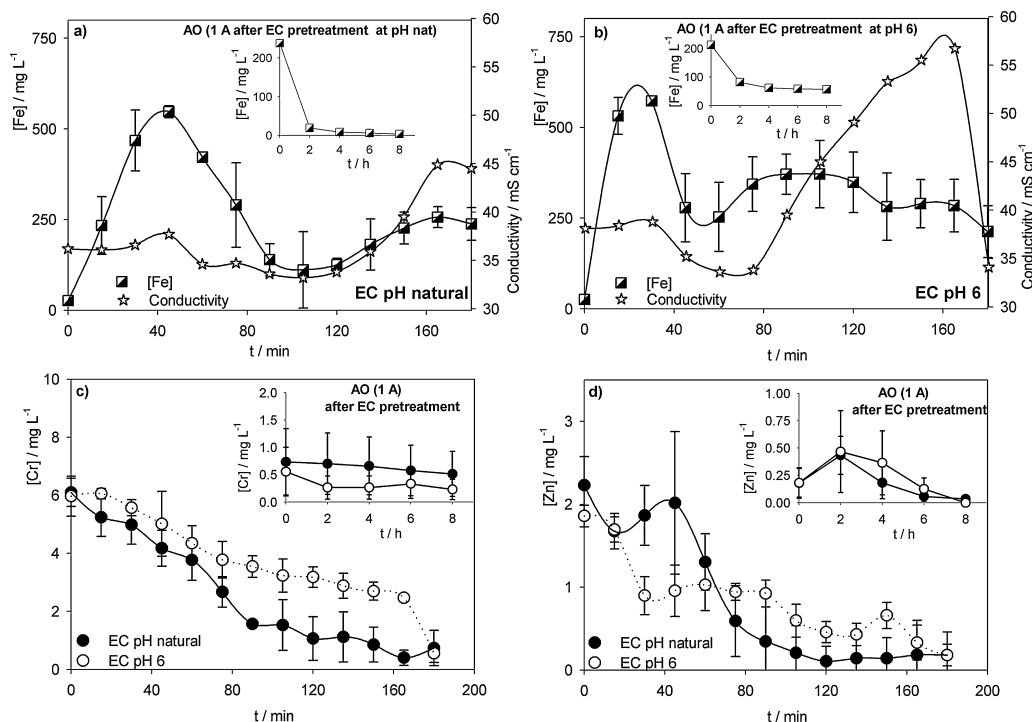
Fig. 6. Variation of the iron concentration and conductivity (a and b) and chromium (c) and zinc (d) concentrations with time for the EC + AO experiments. EC was performed at an initial pH of 6 or the natural pH (8.5), for a 3 h duration, at an applied current intensity of 2.5 A; AO (insets) performed at a current intensity of 1 A, for a 8 h duration. Error bars refer to the standard deviation of the normalized mean values.

load removal rate compared with the assay run at an initial pH of 8.5 and presented a more favorable IEsp. This assay shows the lowest medium specific energy consumption of 11.2 Wh $(g COD)^{-1}$. The least favorable Esp result, 17.5 Wh $(g COD)^{-1}$, was obtained for the combined assay with an initial pH of 6 and 3 h of electrocoagulation. In this case, the extra hour of EC treatment did not lead to a posterior specific energy consumption reduction.

The IEsp values associated with the assays performed at different applied densities were also calculated, and the IEsp values that corresponded to the AO treatment are presented in Fig. 5(b). The energy costs increase with the current density. For the applied intensities of 0.6 and 1 A, the energy consumption decreases with time, showing a higher slope for the lowest intensity. For the highest applied intensity, the IEsp value increases with time, most likely because at this high current intensity, the oxidation of most of the organic compounds in solution are diffusion controlled, leading to a lower current efficiency. For these assays, the medium specific energy consumption increases with the applied current intensity as shown in the following data: 15.4, 21.3 and 21.4 Wh $(g COD)^{-1}$ for the current intensities of 0.6, 1.0 and 1.4 A, respectively.

3.4. Metal ions removal

The variation of the iron, chromium and zinc concentrations during the experiments was determined and is shown in Fig. 6. The variation in iron concentration showed an initial increase followed by a decrease during the EC experiments (Fig. 6a and b). This fluctuation is mainly due to the iron hydroxide formation and posterior precipitation of the suspended/dissolved matter from the leachate, which is followed by shorter, identical cycles. Similar behavior was also reported in the literature [18]. The initial increase in the iron concentration is higher in the assay performed at an initial acidic pH, due to an extra dissolution of the electrode by the acid effect. This effect leads to a shorter time for the decay in iron concentration in solution. Fig. 6a and b shows that the conductivity trend

follows the iron concentration evolution in time for both of the initial pH conditions studied. The results of the variation of the iron concentration during anodic oxidation (insets of Fig. 6a and b) showed an initial sharp decrease followed by a slow, continuous decrease. The correction of the initial pH to 6 leads to a slower removal of the iron from solution. The results of the variation of the chromium and zinc concentrations during EC and AO (Figure 6c, d and insets) showed a decrease in concentration during both processes. The decrease in the chromium and zinc concentrations in EC is due to the simultaneous precipitation with the formed flocs and also due to the reduction in the cathode, with the corresponding deposition of metals on the cathode surface; the concentration decrease in AO is mainly due to the latter process.

4. Conclusions

The application of the combined electrochemical techniques, electrocoagulation and anodic oxidation, may be used to eliminate the recalcitrant organic load from sanitary landfill leachates and/or to increase their biodegradability. For the most favorable experimental conditions applied (3 h of EC at the natural initial pH followed by 8 h of AO at a current density of 700 mA cm^{-2}), an increase in the BOD_5/COD ratio from 0.3 to almost 0.9 was observed, with a 95% COD removal. Simultaneously to the anodic oxidation, cathodic processes can be used to treat the heavy metal content in leachate, because this process reduces the metal ions and deposits them over the cathode. Thus, this combined process can be used to obtain treated leachates, with low content in recalcitrant organic and inorganic compounds, but still with significant organic load, that can be sent to urban wastewater treatment plants comprising biological steps.

The combined electrochemical treatments resulted in very high removals of all forms of nitrogen, although the pH correction during EC did not result in enhanced nitrogen removal. Nevertheless, the lower nitrogen removal during EC is compensated for during

AO and, for total nitrogen, the removal is even higher in the combined process when EC is run at a pH of 6. During EC, chromium was almost completely removed, and zinc was partially removed; the remainder of the zinc was removed during AO. The concentration of iron increases during EC and decreases during AO, and its removal is enhanced with increasing AO applied current density. This initial increase in iron concentration during EC was higher in the assay that was performed at a lower pH due to the extra dissolution of the electrode by the acid effect. For both of the initial pH conditions tested, an iron content fluctuation occurs during EC, mainly due to the cycles of iron hydroxide formation and precipitation. This fluctuation in the iron content is strongly correlated with the variation in the conductivity.

Acknowledgements

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.apcatb.2013.07.054.

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